(12) UK Patent Application (19) GB (11) 2 034 243 A

(21) Application No 7939755
(22) Date of filing 16 Nov 1979
(30) Priority data
(31) 78/44959
(32) 17 Nov 1978
(33) United Kingdom (GB)
(43) Application published
4 Jun 1980
(51) INT CL[3]
D01D 5/42
(52) Domestic classification
B5B 363 CE
(56) Documents cited
GB 2012663A
GB 1464475
GB 1275290
GB 1073741
(58) Field of search
B5B
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(54) Fibrillated synthetic polymer material

(57) To obtain a fibrillated synthetic polymer material with improved modulus and tensile strength, for use *e.g.* as reinforcement in cementitious materials, a high density polyethylene sheet or tape is drawn below its melting point to a draw ratio above 15:1, so as to endow it with a tensile modulus of at least 20GPa, and the molecularly oriented drawn film or tape is then fibrillated by a plurality of cutting elements, *e.g.* cutting pins projecting from the surface of a drum, acting parallel to the direction of draw. The fibrillated net or tape may be chopped to produce fibres of substantially uniform length.

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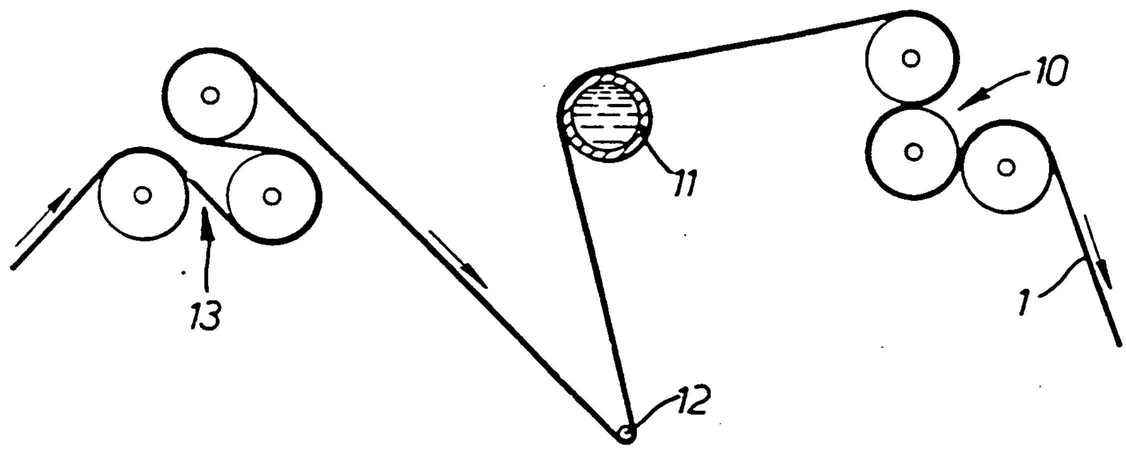

FIG. 1.

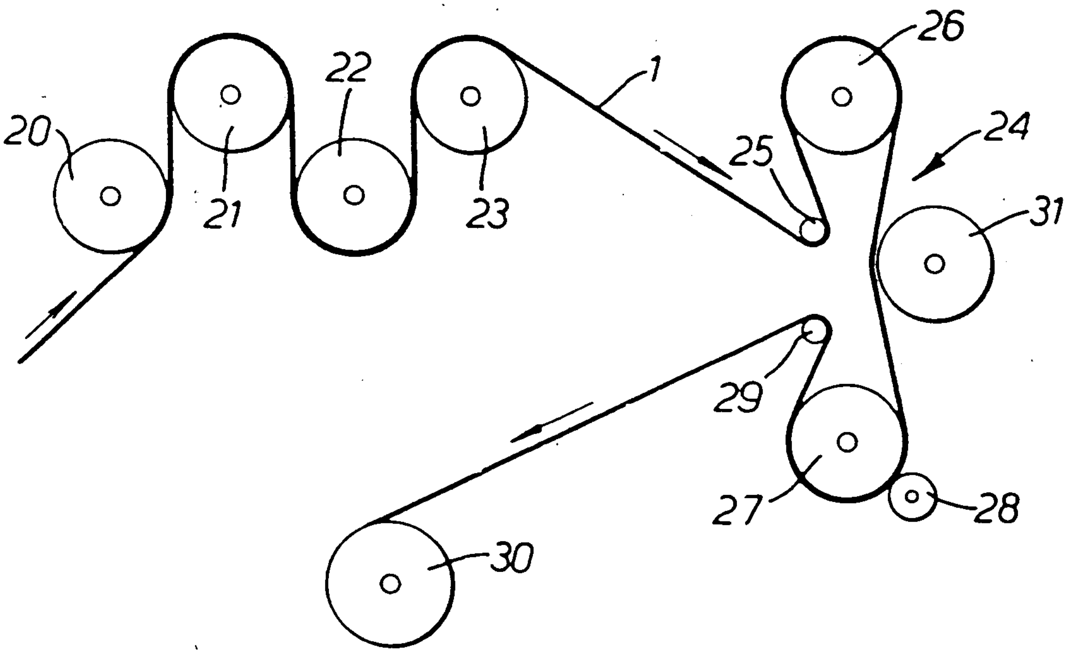

FIG. 2.

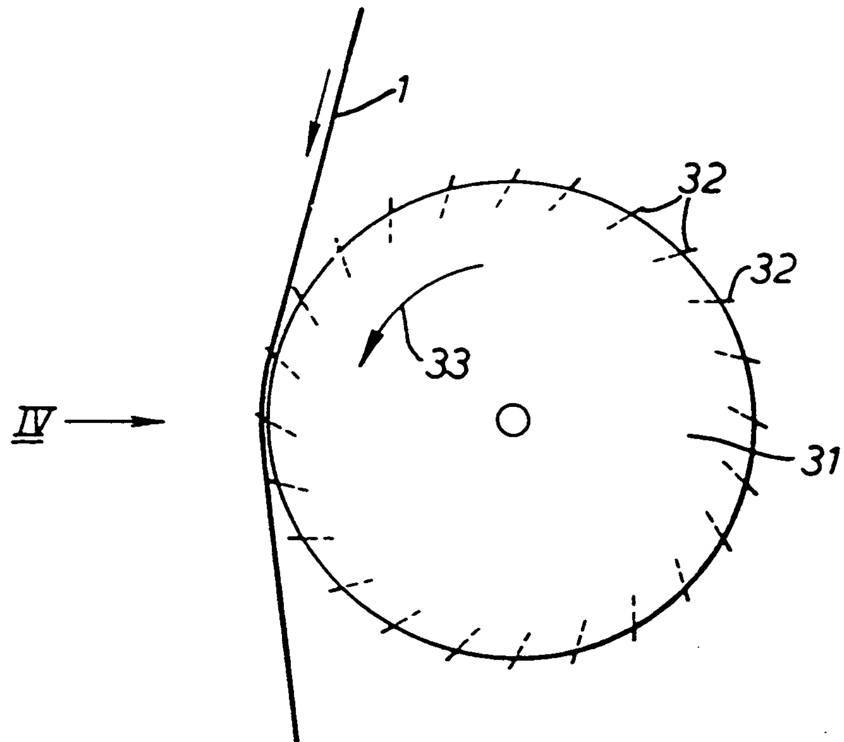

FIG.3.

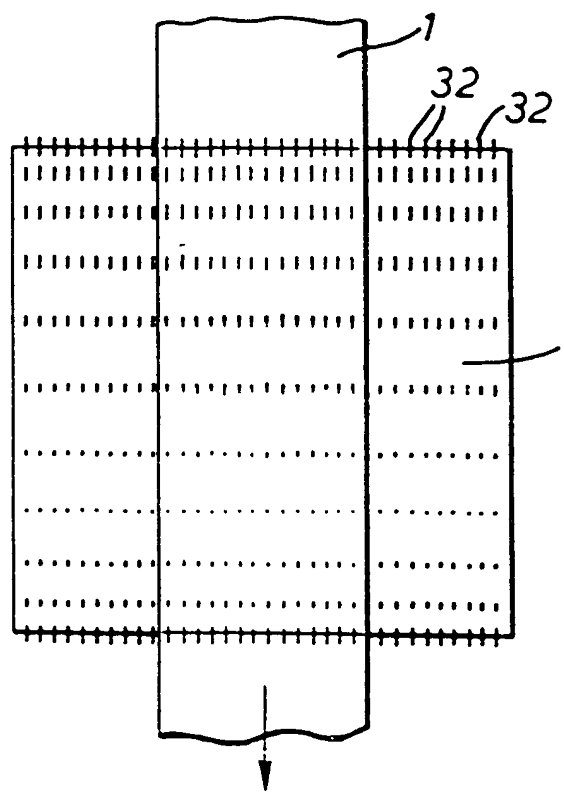

FIG.4.

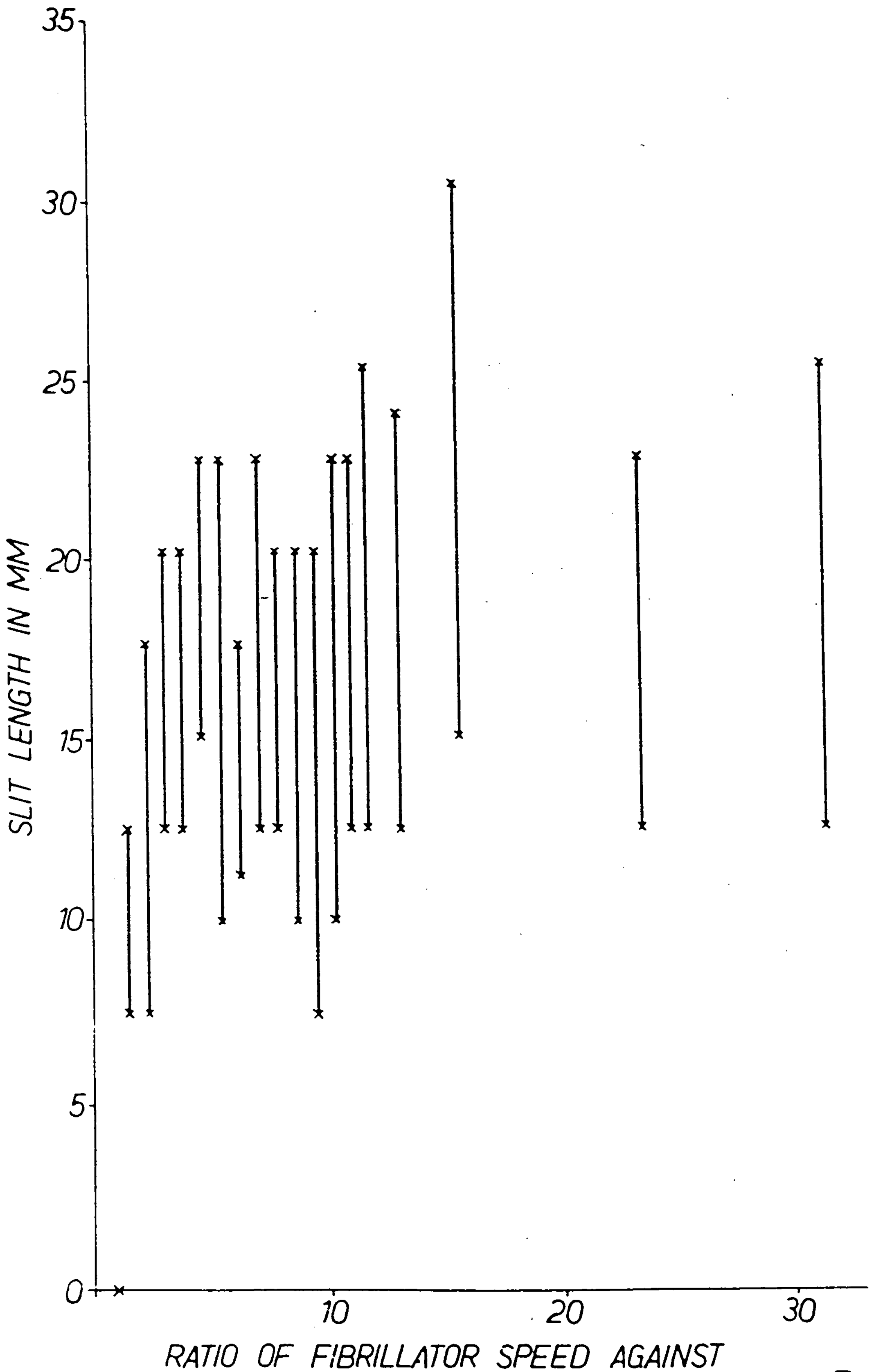
35
30
25
20
15
10
5
0
SLIT LENGTH IN MM
10
20
30
RATIO OF FIBRILLATOR SPEED AGAINST
FEED ROLLER SPEED

FIG. 5.

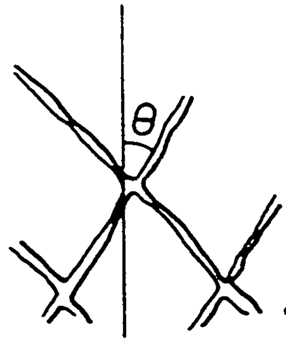

FIG. 6.

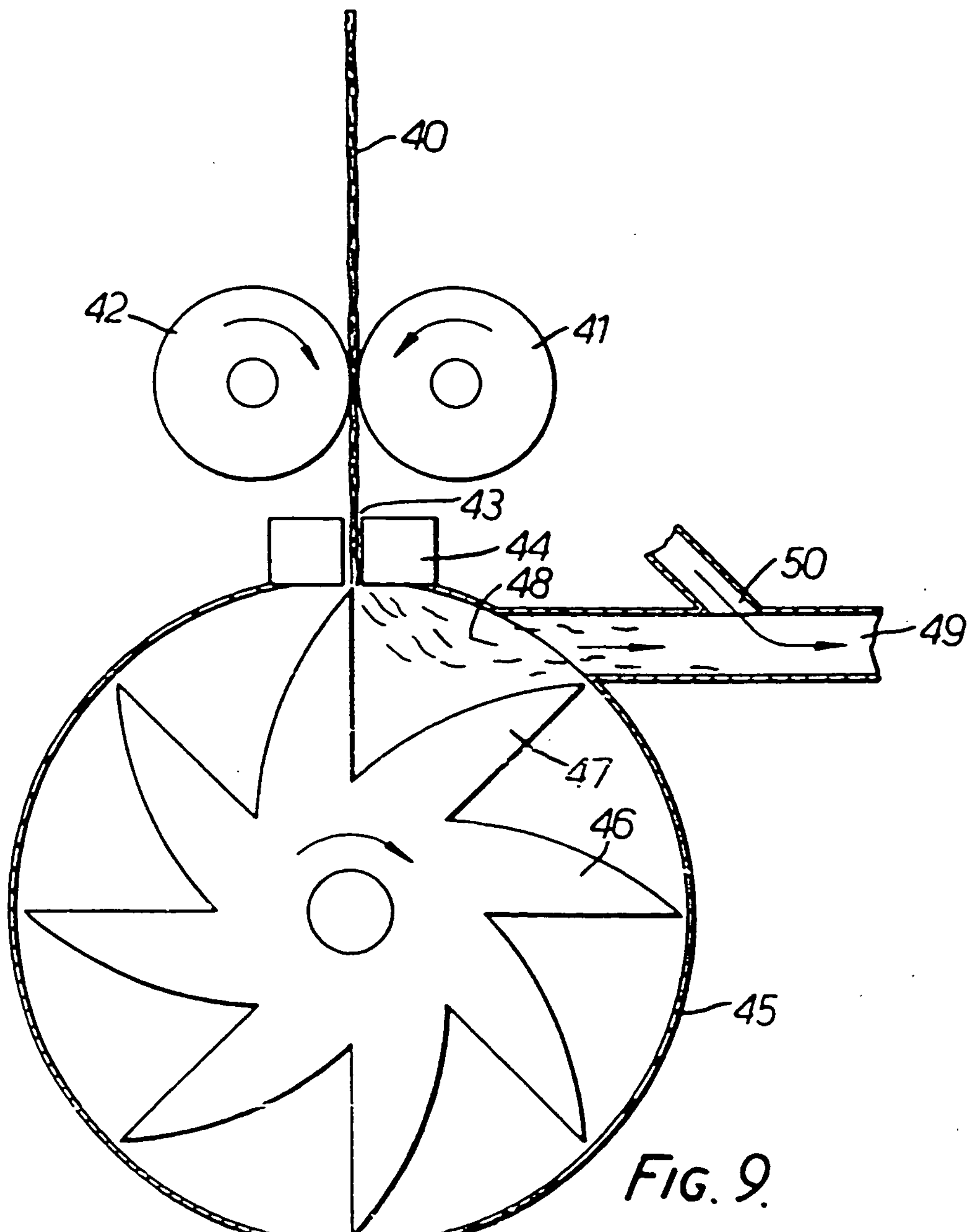

FIG. 9.

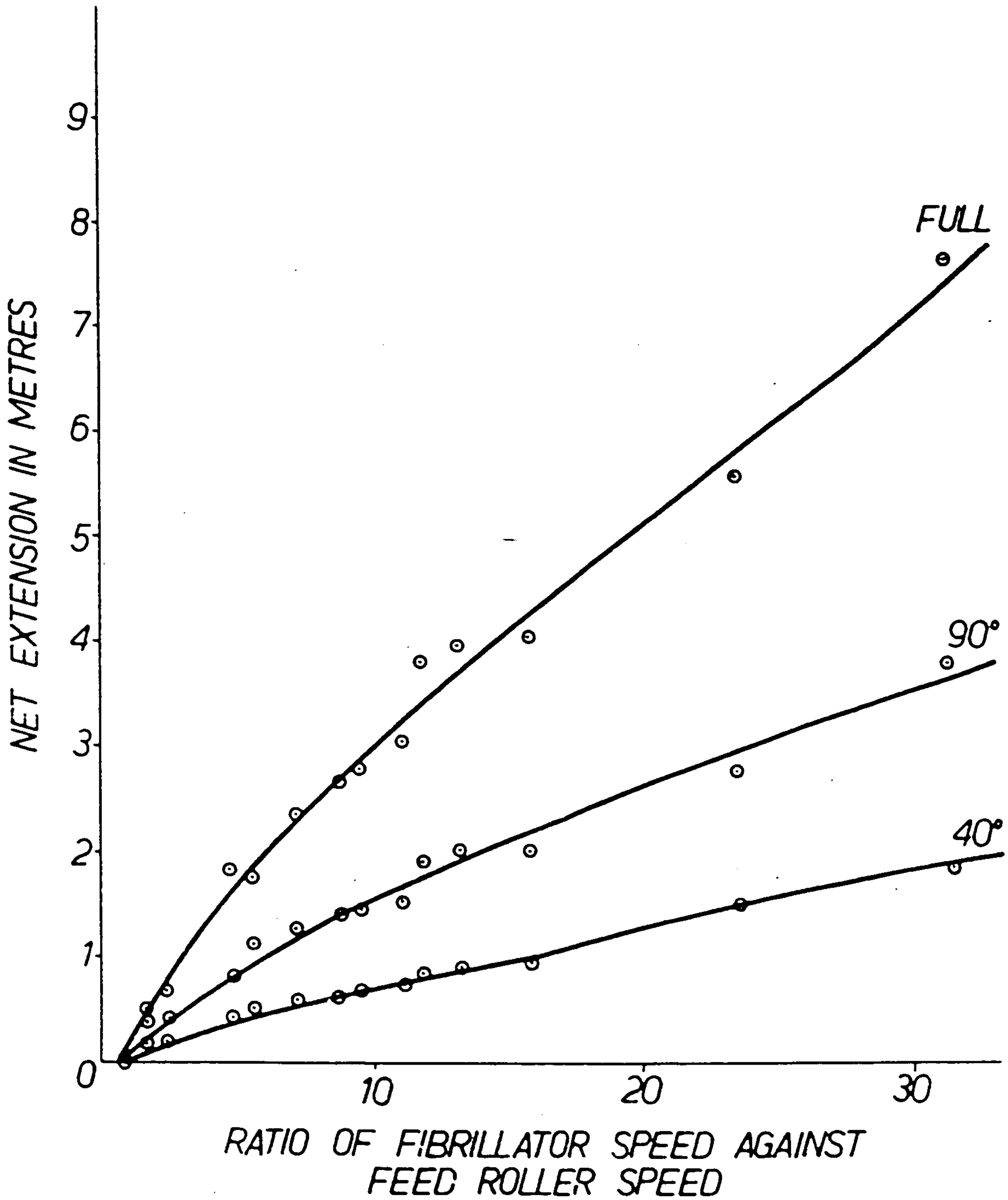
FULL
90°
40°
NET EXTENSION IN METRES
0
1
2
3
4
5
6
7
8
9
10
20
30
RATIO OF FIBRILLATOR SPEED AGAINST
FEED ROLLER SPEED

FIG. 7.

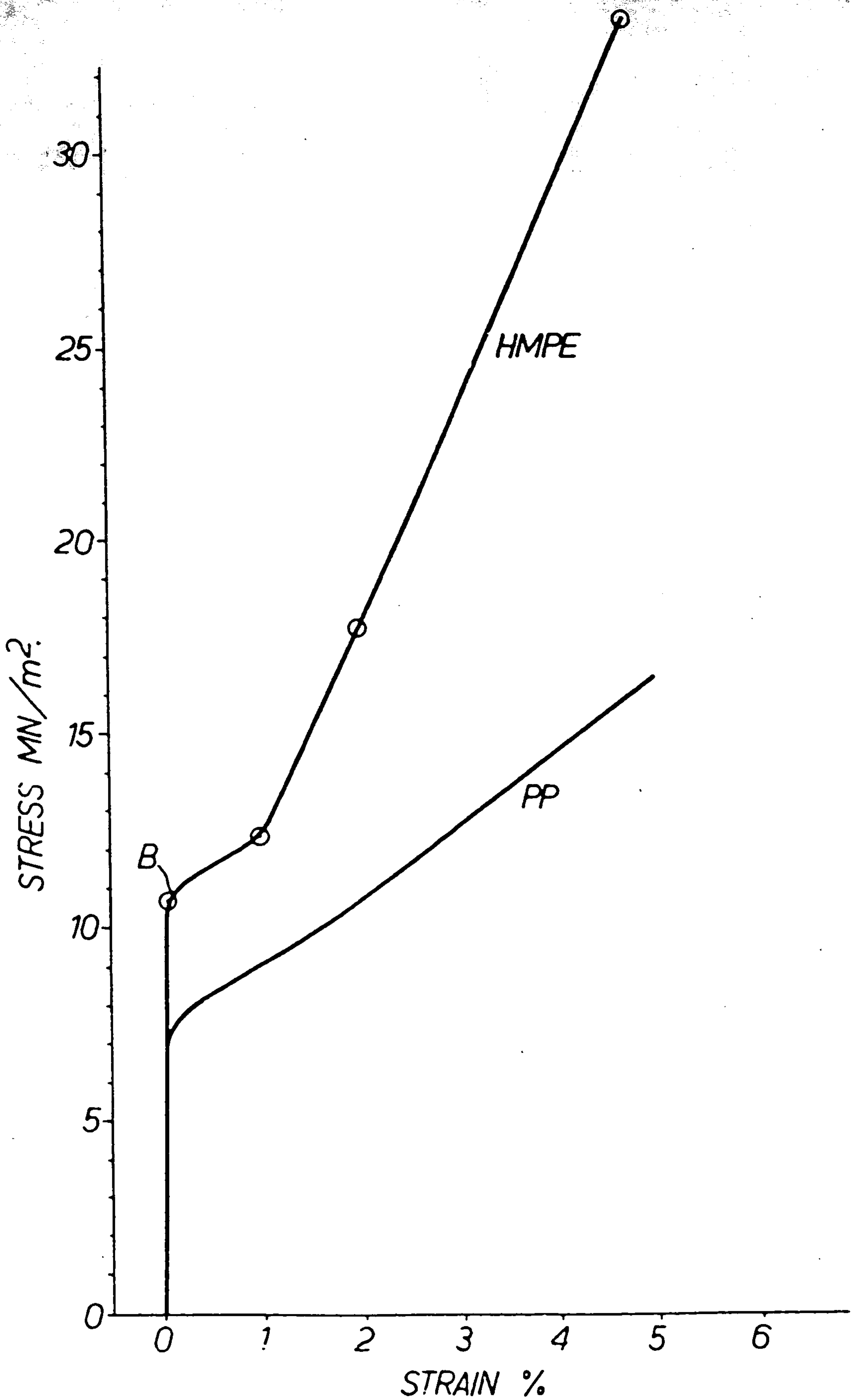

FIG. 8.

SPECIFICATION

Fibrillated synth tic polymer material

This invention relates to methods of producing fibrillated synthetic polym r material and to the fibrillated material thus produced, which may be a coherent fibrillated tape, a net produced by lateral expansion of such a tape or separate fibres produced by disintegration or chopping of a fibrillated tape.

It is known from U.K. Patent Specification No. 1 073,741 to produce a fibrillated synthetic polymer material, e.g. a high density polyethylene, by producing a strip, tube or sheet of the polymer, drawing it over a heated contact plate at a temperature below its melting point to produce a molecular orientation in the strip, tube or sheet and subjecting the strip, tube or sheet to a fibrillating action by running it in contact with a rotating drum which has a plurality of cutting edges or needles projecting from its periphery.

It is also known that, by drawing a sheet or tape of high density polyethylene at a temperature below its melting point to a particularly high draw ratio, above 15:1, it is possible to produce a molecularly oriented film or tape of exceptionally high modulus and high tensile strength parallel to the drawing direction. This drawn material was not previously considered to be usable in a fibrillating process because it has very low strength transverse to the direction of draw and has a tendency to split and disintegrate readily.

An object of the present invention is to produce a fibrillated material with improved modulus and strength as compared with the known fibrillated material referred to above, which will be useful, for example, as reinforcement in composite materials, e.g. in cement or in synthetic resins.

According to the invention, a method of producing a fibrillated synthetic polymer material of high modulus and tensile strength comprises the steps of preparing a sheet or tape of high density polyethylene, drawing the sheet or tape at a temperature below its melting point to a high draw ratio above 15:1 to produce a drawn and molecularly oriented film or tape with a tensile modulus of at least 20GPa and subjecting the drawn film or tape to the fibrillating action of a plurality of cutting elements acting parallel to the direction of draw.

It has surprisingly been found that, in spite of the tendency of the drawn film or tape to split, it can be successfully subjected to the fibrillating process. The product obtained is a tape of interconnected fibres which exhibit high modulus and high tensile strength, as does the tape itself when stressed parallel to the direction of draw and of cutting. If the speed of the cutting elements relative to the drawn film or tape is low, the tape is generally coherent and retains its integrity in normal handling. With a higher relative speed, the tape has a tendency to disintegrate into separate fibres, so that the method of the invention can be used for producing such separate fibres. The tape or the separate fibres can be subjected to a chopping operation by known means to produce relatively short fibres of generally uniform length. When a coherent tape is produced, it can be expanded laterally, i.e. transversely to the direction of draw and of cutting, to form a net.

In carrying out the method of the invention the fibrillating action is preferably produced by a plurality of cutting pins projecting from the surface of a drum, which is mounted so that its periphery intersects the path of the drawn film or tape, the drum being rotated so that the pins contact the film or tape while moving in the same direction as the film or tape but at a higher speed. The film or tape is preferably transported past the pin-carrying drum between respective rollers, the path of the film or tape being such that it embraces the greater part of the periphery of the rollers immediately before and after the pin-carrying drum.

In this specification, the term "high density polyethylene" is used to indicate polyethylene having a density of 0.94 $gm.cm^{-3}$ or higher.

The drawing of the film or tape is preferably carried out in the manner described and claimed in our British Patent No. 1,541,681, using tension rollers and a heated metal tube.

Specific embodiments of the invention will now be described in more detail by way of example and with reference to the accompanying drawing, in which:—

Figure 1 is a diagrammatic elevation of apparatus for carrying out the drawing of the sheet or tape,

Figure 2 is a similar diagrammatic elevation of apparatus for subjecting the drawn film or tape to the fibrillating process.

Figure 3 is a diagrammatic elevation, to a larger scale, of the fibrillating drum acting on the tape,

Figure 4 is a view on the arrow IV of Figure 3.

Figure 5 is a graphical representation of the range of lengths of slit produced in the film or tape by the fibrillator at different relative spe ds of tape and fibrillator,

Figure 6 is a plan view of a portion of net produced by the method of the invention, which has been opened up by expanding it transversely to the direction of the slits produced in the film or tape by the fibrillator, so that the fibres are inclined at an angle θ to the direction of the slits.

Figure 7 is a graphical representation of the variation of the transverse extension of the net achieved at three different fibre inclinations, plotted against the relative speed of tape and fibrillator.

Figure 8 is a graphical representation of tensile stress/strain relationships for samples of cement reinforced with fibres of the invention and with drawn polypropylene fibres for comparison and

Figure 9 is a diagrammatic representation of apparatus for chopping fibres or nets to uniform length.

The device shown in Figure 1, for drawing a tape 1 of high density polyethylene, comprises a frame, not shown, carrying a set of t nsion drawing rollers 10, a small guide pin or roller 12 and a set of feed rollers 13. Betw en the guide pin 12 and the rollers 10 ther is a fixed transverse metal tube 11 through which hot oil can be circulated continuously at a pred termined temperatur by known means (not shown).

The polyethylene tape 1 passes round the heated tube 11 with an angle of wrap-around of between 60° and 90°. The polyethylene is drawn from a neck which is formed at the transverse section of the tape which is heated by the tube 11. To produce the required molecular orientation and a tensil modulus of at least 20GPa, the drawing rollers 10 are driven at such a speed in relation to the feed rollers 13 that the tape is drawn to a draw ratio of above 15:1 and the temperature of the oil in tube 11 is controlled so that drawing is effected at a temperature below the crystalline melting point (about 130°C) of the high density polyethylene. The preferred drawing temperature is in the range 80°C to 120°C, the upper end of this range being preferred when the drawing speed exceeds 10 metres per minute. This process is described in more detail in our prior British Patent Specification No. 1,541,681.

From the apparatus of Figure 1, the tape is supplied to the apparatus shown in Figure 2, being passed around rollers 20, 21, 22 and 23 before passing to the fibrillator indicated generally at 24. The drawn tape 1 passes around a small roller 25 and thence around the greater part of the periphery of a feed roller 26, from which it passes in close proximity to a rotating fibrillator drum 31 and thence to a receiving roller 27 and co-operating nip roller 28. A further small roller 29 is provided to ensure that the film passes around the greater part of the periphery of the receiving roller 27. Thereafter the drawn and fibrillated tape is passed to a wind-up roller 30.

The fibrillator drum 31 is illustrated to a larger scale in Figures 3 and 4. The fibrillator drum 31 is provided with a large number of cutting pins 32 angled backwardly in relation to the direction of movement of the drum (indicated by arrow 33) and projecting by substantially 1 mm from the peripheral surface of the drum, so as to penetrate through the tape 1 and cut a multitude of slits in the tape as it moves past the drum. The pins 32 are set in rows parallel to the axis of the drum, the distance between rows being substantially 6 mm and the distance between adjacent pins in a row being substantially 1 mm. A variable speed drive is provided for rotating the fibrillator drum 31.

In the particular examples now to be described, the high density polyethylene tape employed was that sold by Hoechst Chemicals Limited under the Trade Mark HOSTALEN GD 4760. The temperature of the heated tube 11, which is the drawing temperature of the polyethylene, was 110°C. The drawing speed (i.e. the haul-off speed through tension rollers 10) was 2 metres per minute. The draw ratio produced was 21:1. The dimensions of the drawn tape were 19 mm in width x 0.041 mm in thickness. The secant modulus of the drawn tape at 0.1% strain and 20°C was 32GPa measured at 5% per minute strain rate and the tensile strength was 0.45GPA.

The drawn tape was fibrillated, using the apparatus of Figure 2. Experiments were made in varying the speed of rotation of the fibrillator drum 31 and the speed of the tape 1 past the drum, while in every case ensuring that the pins 32 contacted the tape while moving in the same direction as the tape but at a higher speed. With a tape speed of 12 metres per minute and a peripheral speed of the fibrillator drum of 45 metres per minute, a coarse net fibrillation was achieved. The product was a coherent tape which retained its integrity, when handled normally.

With a larger difference between tape speed and fibrillator speed, e.g. with a tape speed of 3 metres per minute and a fibrillator drum peripheral speed of 82 metres per minute and again with a tape speed of 9 metres per minute and a fibrillator drum peripheral speed of 182 metres per minute, the product was heavily fibrillated, producing a tape with a tendency to disintegrate into individual fibres when drawn apart transverse to the direction of draw and of cutting.

The length of the slits produced in the film or tape 1 by the pins 32 for different ratios of the peripheral speed of the fibrillator drum to the speed of the film or tape was measured, with the results which are shown graphically in Figure 5. The slit length varies over a range in all cases. At low ratios of fibrillator speed to film speed, the slits tend to be shorter, but with ratios above 3:1 little difference in slit length is evident.

Figure 6 illustrates a portion of a net produced by the method of the invention, showing how the drawing apart or expansion of the fibrillated film or tape transverse to the direction of the slits reveals its net form. As the tape is expanded transversely, the angle θ between the individual fibres and the direction of the slits increases. Measurement of the transverse extension of the net at two different values of θ, namely 40° and 90° and at the maximum transverse extension achieveable, on samples of fibrillated film or tape which had been operated on at different ratios of fibrillator speed to tape speed, showed that the extension increased more or less linearly with the speed ratio, as illustrated in Figure 7, presumably due to the greater number of slits produced in a given area of the film or tape as the speed ratio increased.

Products made in accordance with the inv ntion from three different samples of high density polyethylene were subjected to testing for modulus and tensile strength of the fibrillated tape in the direction parallel to the direction of draw and of cutting, with the following results. Figures for the tap

prior to fibrillation are included for comparison.

Tape				Befor Fibrillation		After Fibrillation	
Sample	Width mm	Thickness mm	Draw Ratio	Tensile Modulus GPa	Tensile Strength GPa	Tensile Modulus GPa	Tensile Strength GPa
1	19.0	.040	20.1	32.3	0.45	31.2	0.45
2	25.4	.038	13.1	37.0	0.46	35.0	0.45
3	20.3	.036	20.1	47.0	0.48	46.5	0.48

It will be seen that the fibrillated polyethylene tape, when stressed parallel to the direction of draw and of cutting, retains the high moduluis and tensile strength of the drawn polyethylene tape, which are very considerably higher than the corresponding figures for the known polypropylene product.

Samples of fibre-reinforced cement composites were made up incorporating fine nets of fibrillated high density polyethylene made according to the invention and further samples were made up with fibrillated polypropylene nets for comparison.

The composition of the composites was

Cement Matrix:	Parts by Weight
Ordinary Portland cement	1
Pulverised fuel ash	0.25
Sand (fine)	0.19
Water	0.34
Plasticiser (sold under trade name MELMENT)	0.025

The fibrillated nets were produced from drawn tapes as described above, using a speed ratio of fibrillator drum to tape of 2.36, the fibrillated tape being expanded to an angle θ of 40°.

The fibrillated nets, amounting to 8% by volume of the composites, were incorporated by hand lay-up in test specimens whose dimensions were 300 mm x 25 mm x 6 mm and the specimens were cured for 28 days under water at 20°C. Tensile tests of the specimens were then carried out on an Instron testing machine at 20°C at a low strain rate of approximately 10% per minute.

The results are shown graphically in Figure 8, in which the curve marked HMPE illustrates the results using the high modulus polyethylene and that marked PP the results with polypropylene nets. It will be seen that the samples containing fibrillated polyethylene nets made in accordance with the invention depart from proportionally (at the "bend-over point" B) at higher stress levels and the slope of the curve is steeper, showing that the samples are stiffer than those reinforced with polypropylene nets.

It is also possible to chop fibrillated tapes or nets produced according to the invention, using chopping apparatus of a known kind as illustrated in Figure 9, to produce relatively short fibres of generally uniform length. The fibrillated tape 40 is fed by means of feed rollers 41, 42 to a slot 43 in a cutting head 44 situated in the periphery of a cylindrical cutter housing 45. A rotary cutter 46 is journalled on the axis of housing 45 and has a plurality of equally-spaced cutting arms 47 whose tips pass in close proximity to the end of the slot 43, so as to chop the tape 40 issuing therefrom into fibres 48 of substantially uniform length. The fibres 48 are removed *via* an outlet 49 by means of an air jet extractor 50.

In addition to the use of the fibrillated products as reinforcement in cement composites describ d above, they may also be employed, on fibrillated tapes or nets or as fibres, in synthetic resin composites

CLAIMS

1. A method of producing a fibrillated synthetic polymer material of high modulus and tensile strength, comprising the steps of preparing a sheet or tape of high density polyethylene, drawing the sheet or tape at a temperature below its melting point to a high draw ratio above 15:1 to produce a drawn and molecularly oriented film or tape with a tensile modulus of at least 20GPA, and subjecting the drawn film or tape to the fibrillating action of a plurality of cutting elements acting parallel to the

direction of draw.

2. A m thod according to Claim 1, wherein the fibrillating action is produced by a plurality of cutting pins projecting from the surface of a drum, which is mounted so that its periphery intersects the path of the drawn film or tape, the drum being rotated so that the pins contact the film or tape while moving in the same direction as the film or tape but at a higher speed.

3. A method according to Claim 2, wherein the film or tape is transported past the pin-carrying drum between respective rollers, the path of the film or tape being such that it embraces the greater part of the periphery of the rollers immediately before and after the pin-carrying drum.

4. A method according to any one of the preceding claims, wherein the fibrillated film or tape is chopped into fibres of substantially uniform length.

5. A method according to Claim 4 wherein the chopping is effected by a rotary chopping apparatus having a rotary cutter with a plurality of equally spaced cutting arms arranged to act on the fibrillated film or tape as it is fed into a housing of the cutting apparatus.

6. A method of producing a fibrillated synthetic polymer material of high modulus and tensile strength, substantially as hereinbefore described with reference to the accompanying drawings.

7. A fibrillated high density polyethylene material produced by the method of any one of the preceding claims.

Printed for Her Majesty's Stationery Office by the Courier Press, Leamington Spa, 1980. Published by the Patent Office, 25 Southampton Buildings, London, WC2A 1AY, from which copies may be obtained.